भारतीय मानक
Indian Standard

IS 1448 (Part 120) : 2021

पैट्रोलियम और इसके उत्पादों के परीक्षण की पद्धितियाँ

भाग 120 स्नेहक तेल में जिंक

(पहला पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 120 Zinc in Lubricating Oil

(First Revision)

ICS 75.100

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भारतीय मानक ब्यूरो
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June 2021 **Price Group 2**

Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This Standard was originally published in 1986 and was adopted with permission from Institue of Petroleum Standard No. IP 117/82.

In first revision of this standard, clause for references have been included.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

PART 120 ZINC IN LUBRICATING OIL

(First Revision)

1 SCOPE

This Indian Standard (Part 120) is intended for the determination of zinc in unused lubricating oil. It separates zinc from magnesium and alkaline earth metals, but there are chances of interference by other heavy metals.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

IS No.	*Title*
1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
264 : 2005	Nitric acid — Specification (*third revision*)
265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
266 : 1993	Sulphuric acid — Specification (*third revision*)

3 SUMMARY OF METHOD

The sample is ashed and the zinc determined as zinc hydroxyquinolate.

4 REAGENTS

4.1 Sulphuric Acid — Concentrated, relative density 1.84 (*see* IS 266).

4.2 Nitric Acid — Concentrated, relative density 1.42 (*see* IS 264).

4.3 Hydrochloric Acid — 2 N approximate (*see* IS 265).

4.4 Thymol Blue Indicator — Dissolve 0.1 g thymol blue in 50 ml ethyl alcohol and dilute to 100 ml with water.

4.5 Ammonium Hydroxide — Concentrated, relative density 0.880.

4.6 Oxine Solution — Dissolve 2 g of 8-hydroxyquinoline in 10 ml glacial acetic acid and dilute to 100 ml with water.

4.7 Ammonium Acetate Buffer — Saturated aqueous solution.

5 APPARATUS

5.1 Silica Crucible

5.2 Muffle Furnace

5.3 Whatman No. 42 or Similar Filter Paper

5.4 Hot Plate

5.5 No. 3 Sintered Glass Filter (20 to 30 µm Porosity)

5.6 Drying Oven

6 PROCEDURE

6.1 Weigh to the nearest 0.01 g into a silica crucible an amount of sample containing 20 to 40 mg of zinc and heat it gently until the oil ignites. Allow it to burn until a coke residue is formed, maintaining the burning with a gas flame if necessary; and continue heating with a gas flame until all the volatile matter has been driven off. Transfer the crucible to a muffle furnace at 550 °C and heat it until the ash is free from all visible traces of carbon.

6.2 Cool, moisten the residue with approximately 2 ml of concentrated sulphuric acid. Heat the crucible until white fumes are evolved and assist the oxidation by the addition of concentrated nitric acid. Heat again until white fumes are evolved.

> *Caution* — The concentrated nitric acid shall be added drop-wise and slowly in order to avoid an excessively violent reaction.

6.3 Allow the crucible to cool, add 10 ml of 2 N hydrocloric acid cautiously, and transfer the solution to a 250 ml beaker using approximately 100 ml of water. Heat until the solution just boils and allow it to stand for at least 4 h, or preferably overnight.

6.4 Filter through Whatman No. 42 or similar filter paper, wash with hot water, and add two drops of thymol blue indicator. Add concentrated ammonium hydroxide, drop-wise with stirring, until the solution just changes to blue, then add 2 N hydrochloric acid drop-wise until the solution is yellow.

6.5 Heat the solution to 70 °C and add 10 ml of oxine solution for each 10 mg of zinc present. Then add 5 ml of amonium acetate buffer solution. Stir the solution, remove from the hot plate and stand for approximately 15 min. Filter the precipitate on a weighed No. 3 sintered glass filter (20 to 30 pm porosity). Wash thoroughly with hot water and dry the precipitate to constant mass at 130 to 140 °C.

7 CALCULATION

7.1 Calculate the percentage of zinc in the sample from the equation:

$$\text{Zinc, percent by mass} = \frac{18.49 \times M_1}{M_2}$$

where

M_1 = mass of precipitate, in g; and
M_2 = mass of sample, in g.

7.2 Report the result to the nearest 0.001 as zinc content, percent by mass.

8 PRECISION

Results of duplicate test shall not differ by more than the following amount:

Mean	*Repeatability*	*Reproducibility*
X	0.036 (X + 0.025)	$0.33\sqrt{X}$
0.02	0.001	0.004
0.05	0.002	0.007
0.1	0.004	0.010
0.2	0.008	0.014

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ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Sampling and Test for Petroleum, its Products,
Gaseous Fuels and Lubricants Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray (***Chairman***)
Air Headquarters, Ministry of Defence, New Delhi	Wg Cmdr S. A. Tawab Wg Cmdr D. Anandraj Roy (*Alternate*)
All India Instrument Manufacturers and Dealers Association, Mumbai	Prof G. M. Rao
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Bharat Petroleum Corporation Limited, Mumbai	Shri C. Shanmuganathan Shri R. Subramanian (*Alternate*)
Castrol India Limited, Mumbai	Shri Raman Rai
Central Institute of Plastics Engineering and Technology, Bhuwaneshwar	Dr Smita Mohanty
Central Revenue Control Laboratory, New Delhi	Dr T. A. Sreenivasa Rao
Chennai Petroleum Corporation Limited, Chennai	Shri H. Ramakrishnan Dr V. Selvavathi (*Alternate*)
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Shri P. K. Singh Shri S. R. K. Rao (*Alternate* I) Shri S. Dutta (*Alternate* II)
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Directorate General of Quality Assurance, Ministry of Defence, New Delhi	Shri C. B. Pandey
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Hindustan Petroleum Corporation Limited, Mumbai	Shri Madhusudan Shri Elecheran Kumar (*Alternate* I) Shri Shitanshu Pati (*Alternate* II)
HPCL Mittal Energy Limited, Noida	Shri Narendra Kumar Gupta Shri Satyabrata Bandyopadhyay (*Alternate*)
Indian Oil Corporation (R and D Centre), Faridabad	Shri V. Kagdiyal Dr S. K. Majumdar (*Alternate*)
Indian Oil Corporation Limited - Refineries and Pipelines Division, New Delhi	Dr Ashutosh Mishra Shri Shashi Pal Singh (*Alternate*)
Indian Oil Corporation Limited, Mumbai	Shri P. D. Dusane Shri G. K. Sharma (*Alternate*)
Lubrizol India Limited, Mumbai	Shri Sharad Tarsode

Organization	*Representative(s)*
Mangalore Refinery and Petro Chemical Limited, Mangalore	Shri Shalina Kumar Shri Yogeesha (*Alternate* I) Shri R. M. Prakash (*Alternate* II)
Ministry of Railways, Lucknow	Shri Rajesh Srivastava Shri Sonam Gupta (*Alternate*)
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Numaligarh Refinery Limited, Golaghat	Shri Anjan Kalita Shri Sobhan Konwar (*Alternate*)
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Oil India Limited, Duliajan	Representative
Petroleum and Natural Gas Regulatory Board, New Delhi	Shri Anil Meghani Shri Noor Khan (*Alternate*)
Petroleum Conservation Research Association, New Delhi	Shri M. P. Bangwal
Reliance India Limited, Mumbai	Shri Pramod Mall
Shriram Institute for Industrial Research, Delhi	Shri Shambhu Thakur
Indraprastha Gas Limited, New Delhi	Representative
BIS Director General	Shri N. K. Bansal, Scientist ‘F’ and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shrimati Nagamani T.
Scientist ‘E’ (PCD), BIS

Bureau of Indian Standards

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Amendments Issued Since Publication

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